DESIGN, FABRICATION AND CHARACTERIZATION OF AN IN-PLANE AFM PROBE WITH ULTRA-SHARP SILICON NITRIDE TIP

E. Sarajlic [1], J. Geerlings [2], J.W. Berenschot [2], M.H. Siekman [1,2], N.R. Tas [2] and L. Abelmann [2]

[1] *SmartTip, Enschede, The Netherlands*
[2] *MESA+ Research Institute, University of Twente, PO Box 217, 7500 AE Enschede, The Netherlands*

***Abstract* — Scanning rates of the atomic force microscope (AFM) could be significantly increased by integrating the force sensing probe with microelectromechanical systems (MEMS). We present a micromachining method for batch fabrication of in-plane AFM probes that consist of an ultra-sharp silicon nitride tip on a single crystal silicon cantilever. Our fabrication method is fully compatible with the silicon-on-insulator (SOI) micromachining allowing a straightforward monolithic integration of the AFM probes with high-aspect-ratio monocrystalline silicon MEMS. Scanning probes with a sharp tip having diameter of less then 10 nm are successfully realized and tested in a commercial AFM set-up demonstrating feasibility and the large innovation potential of this method.**

***Keywords:* Atomic Force Microscopy, Probes, KOH etching, Video-rate AFM**

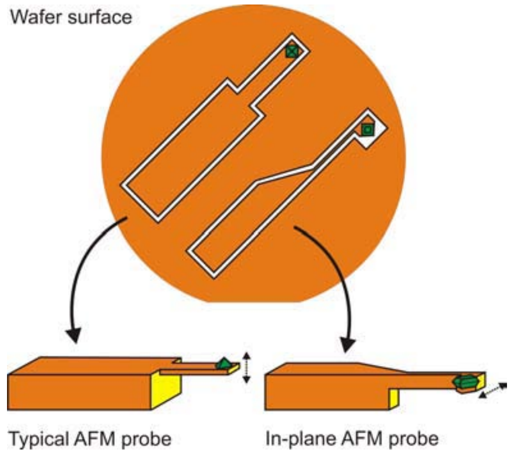


Figure 1: *Typical AFM probe with out-of-plane tip and lateral cantilever with in-plane tip.*

I – Introduction

In atomic force microscopy (AFM) a strong drive exists towards video-rate imaging. In addition to convenience, the video rate AFM will enable observation of many real time dynamic processes that are currently impossible to study e.g. diffusion of individual atoms, atom clusters or molecules, film growth or catalytic reactions [1].

In a conventional AFM system the motion in the vertical z-direction is fastest and requires the highest frequency components. Therefore, the mechanical resonance frequencies of a cantilever force-sensor [2] and the driving piezoelectric element [3] are the main speed limiting factors in the conventional AFM systems. In order to obtain higher scanning rates, the external piezoelectric actuation can be substitute with a micromechanical actuator having an integrated force-sensing element [4]. Small dimensions and extremely small mass of the microactuators will improve vibration isolation and enable higher scanning rates.

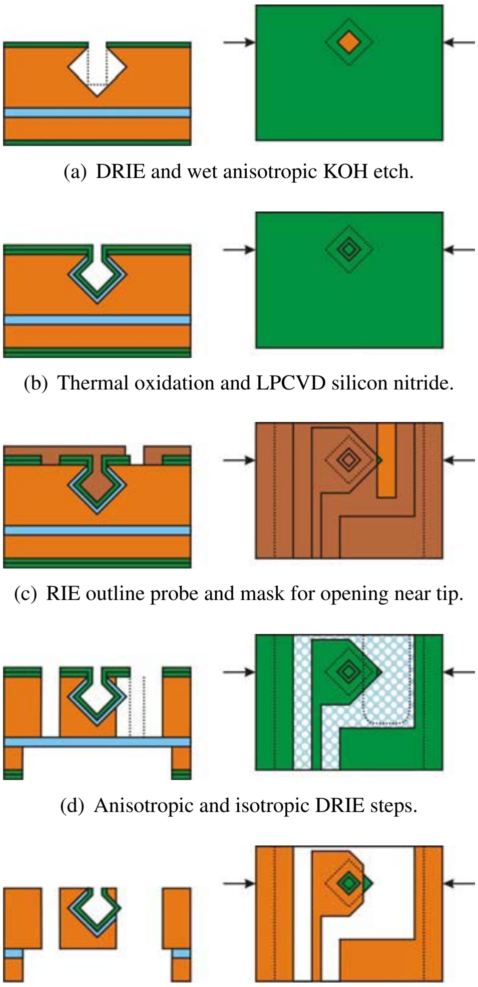
In this paper we present a novel micromachining method for bulk fabrication of AFM probes with an ultra-sharp tip. This method, which is fully compatible with silicon-on-insulator (SOI) micromachining, allows for easy integration of the scanning probes with high-aspect-ratio monocrystalline silicon microactuators. Distinguishing characteristics of our process are as follows: (i) It allows fabrication of the AFM cantilevers with an in-plane tip (see Figure 1). In the in-plane configuration, the oscillation direction of the cantilever is parallel to the wafer plane and coincides with the preferable motion direction of different electrostatic microactuators (e.g. parallel plate actuators or comb drives) allowing their straightforward integration. Furthermore, the resonance frequency of the in-plane cantilever probes can be more precisely controlled because both length and thickness are controlled by lithography. Therefore the resonance frequency is less dependent on the process parameters. (ii) The process results in a monocrystalline silicon cantilever. Superior mechanical properties of single crystal silicon such as practically no stress and low intrinsic damping [5] will lead to improved probe performance. (iii) An important innovative aspect of our method is the use of silicon moulds obtained by KOH etching to form in-plane silicon nitride tips. Silicon nitride, which is a wear resistant material will result in the improved life time of the probes. (iv) The in-plane tips are ultra-sharp allowing for high resolution imaging because the resolution of an AFM set-up is mainly determined by the tip sharpness of the probe.

Outline of this paper is as follows. In Section II we describe the basic steps of the microfabrication process and present two different methods for the fabrication of in-plane tips. In Section III we give details on the design of the first probe prototype. The fabrication results and experimental characterization of the probes is presented in Section IV. Conclusions are drawn in the last section.

II – In-plane AFM probe

A. Microfabrication process

The fabrication process, schematically shown in Figure 2, is based on an SOI wafer with a (100) top silicon layer.



(a) DRIE and wet anisotropic KOH etch.

(b) Thermal oxidation and LPCVD silicon nitride.

(c) RIE outline probe and mask for opening near tip.

(d) Anisotropic and isotropic DRIE steps.

(e) RIE top layer silicon nitride and oxide etch.

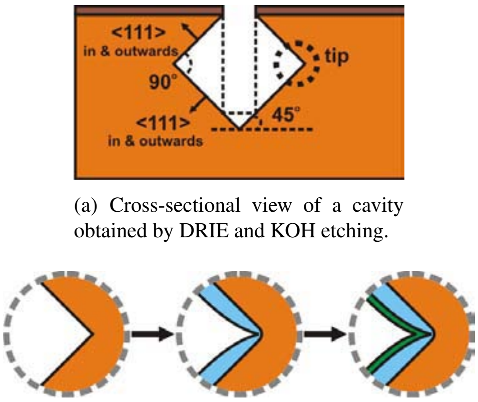
Figure 2: *SOI compatible fabrication process of in-plane AFM probes.*

In the top layer, a cavity is etched by combination of deep reactive ion etching (DRIE) and wet anisotropic etching in KOH (a). After etching of the cavity, thermal oxidation is performed followed by LPCVD deposition of silicon nitride (b). Next, the outline of the scanning probe is defined in the silicon nitride/silicon oxide stack by reactive ion etching (RIE). Subsequently, an opening near the probe tip is lithographically defined in a photoresist layer (c) and the tip is partially released by combination of anisotropic and isotropic dry silicon etching in a DRIE plasma system. After the tip release, the photoresist mask is removed and the probe layout is etched by DRIE using the silicon nitride/silicon oxide stack as the etching mask. Subsequently, a back etch is preformed (d). Next, the top silicon nitride layer is removed by a blanket RIE step. Finally, the probe is released by etching of silicon oxide (e). The process results in a monocrystalline silicon scanning probe with an in-plane silicon nitride tip.

B. Tip formation

In our process, the probe tip is formed by refilling a silicon mould with a silicon nitride layer. The sharpness of the probe tip is defined by the profile of the mould. In this paper, we devised and tested two innovative methods to obtain sharp in-plane tips.

In the first method, which is shown in Figure 3, an octahedron is used as a mould. The silicon octahedron is formed by directional plasma etching followed by wet anisotropic etching in aqueous KOH solution. The in-plane tip is formed in one of the corners of the octahedron, which are sharpened by thermal oxidation prior to silicon nitride deposition. This method results in a plane-symmetrical tip with four faces defined by <111> crystallographic planes of the silicon octahedron. In order to obtain a single sharp tip a perfect symmetrical octahedron is required.



(a) Cross-sectional view of a cavity obtained by DRIE and KOH etching.

(b) Tip sharpening by oxidation.

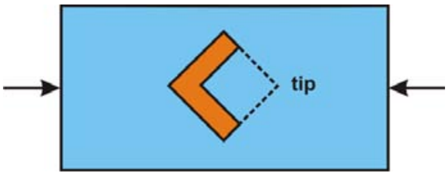
Figure 3: *Four-face tip formation.*

In the second method, illustrated in Figure 4, a pyramidal silicon mould is formed using an L-shaped mask opening. Silicon oxide is used as the masking layer. The tip formed in the top corner of the mould is bounded by two <111> planes of the silicon pyramid and the top plane of silicon oxide. As the three planes always intersect in a single point, we expect that this design will results in an ultra-sharp tip.

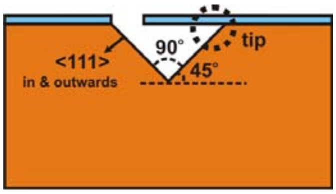
III – Design

To demonstrate the new fabrication method two different types of probes were designed. AFM probes with four-face nitride tips and probes with three-face nitride tips. The layout of silicon cantilever and holder block are the same for both type of probes.

When the tips are formed (as described in the previous section), the next step is the formation of the block



(a) Topview L-shape mask.



(b) Cross-sectional view of a cavity obtained by KOH etching.

(c) Tip sharpening by oxidation.

Figure 4: *Three-face tip formation.*

and cantilever that holds the tip (see right side of Figure 1). Because (100) wafers are used, the octahedron (after KOH) is aligned to the flat of the wafer. However, the tip of the octahedron must be perpendicular to the cantilever. Because oblique lines are difficult to obtain (as mask writers have a limited step resolution) the wafer must be rotated 45 degrees clockwise during the lithography steps needed to form the cantilever and holder block.

Initially the cantilever width is equal to the thickness of the wafer. To reduce the width of the cantilever, a backside etch step is applied. The depth of the backside etch therefore determines the width of the cantilever. When a SOI wafer is used, the oxide layer serves as etch stop layer. In this design a width of 40-50 µm was aimed for. The thickness of the cantilever is chosen equal to the minimal accurate thickness that can be obtained, which is 3 µm in this case. Length of the cantilever is calculated from the desired resonance frequency. For AFM tapping mode the resonance frequency must be between 100 and 300 kHz and for contact mode between 5 and 10 kHz. Table 1 summarizes the chosen cantilever dimensions and calculated approximate resonance frequencies. Definitions of the dimension variables are shown in Figure 5.

Because the resonance frequency does not depend on the width of the cantilever, the back etch step is not critical for the resonance frequency. The cantilever is connected to a block with length of 3000 µm, thickness 450 µm and width determined by the thickness of the wafer (380 µm in this case). This block is necessary for handling the probe and placing it in the AFM machine.

Table 1: *Probe dimensions and calculated resonance frequencies of the cantilever.*

	Probe 01	Probe 02	Probe 03	Probe 04
Cantilever				
Length (L_C)	200 µm	300 µm	400 µm	500 µm
Thickness (T_C)	3 µm	3 µm	3 µm	3 µm
Width (W_C)	50 µm	50 µm	50 µm	50 µm
Resonance (f_0)	108 kHz	48 kHz	27 kHz	17 kHz
Holder block				
Length (L_B)	3000 µm	3000 µm	3000 µm	3000 µm
Thickness (T_B)	450 µm	450 µm	450 µm	450 µm
Width (W_B)	380 µm	380 µm	380 µm	380 µm

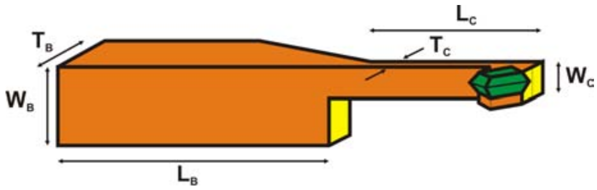


Figure 5: *In-plane probe with definition of variables.*

The holder block with cantilever is connected to the wafer by means of a breakout beam.

IV – Results and Discussion

A. Four-face tip design

Figure 6 shows SEM images of the fabricated in-plane AFM probe with four-face tip.

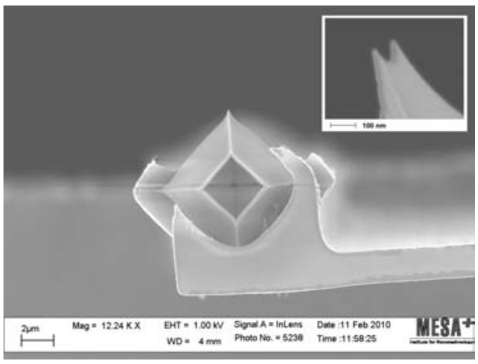


Figure 6: *SEM pictures of the silicon nitride in-plane AFM tip (four-face) on a mono crystalline cantilever. Tip diameter (see inset) is 24 nm (left part of the double tip).*

Clearly visible is the double tip in Figure 6 which indicates that during KOH etching a ridge was formed in the tip corner, instead of a single sharp tip. This invalidates the initial assumption that the four faces of the octahedron (which bound the tip) always intersect in one point, regardless of the shape of the starting cavity. The role of the starting cylinder on the final cavity was studied by KOH simulations in simulation program ACES [6].

Both graphical analysis and simulations of the octahedron formation showed that a misalignment between bottom plane and top plane of the starting cylinder causes the four planes not to intersect in one point. This is also the case when the bottom plane is not completely flat. Because DRIE etching is not perfectly uniform, a perfect cylinder can not be obtained. The four faces of the octahedron will therefore always lead to a ridge instead of a tip. This was practically confirmed during inspection of the fabricated tips in the SEM (shown in Figure 6).

B. Three-face tip design

Because three non-parallel planes always intersect in one single point, this design will result in probes with single tips. In Figure 7 the fabrication results of the three-face tip design is shown. Ultra-sharp single tips were indeed observed during SEM inspection of the probes (see Figure 7).

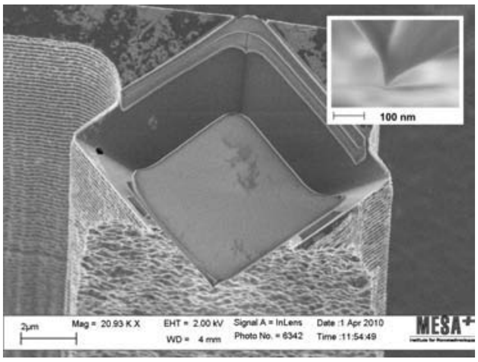


Figure 7: *SEM pictures of the nitride in-plane AFM tip (three-face) on a mono crystalline cantilever. Tip diameter (see inset) is 9 nm.*

On several cantilevers a reflection coating was deposited by evaporation. This coating consisted of a 5 nm Cr layer and a 15 nm Au layer. These probes were mounted in a Nanoscope Dimension 3100 [7] to obtain AFM images. An AFM image (in tapping mode) of a silicon wafer (with native oxide) is shown in Figure 8. A three-face probe was used for this image. Features less than 10 nm can be observed, indicating the tip sharpness.

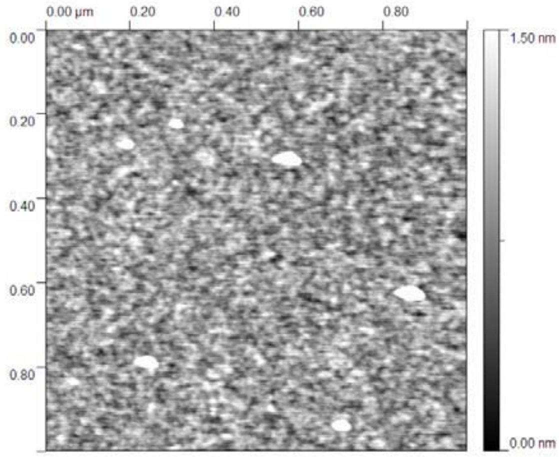


Figure 8: *AFM image of a silicon wafer obtained with a three-face tip probe in tapping mode at 34 kHz.*

V – Conclusions

A new batch manufacturing process for in-plane AFM probes was presented. By using a silicon mould ultra-sharp durable nitride tips can be constructed on a monocrystalline silicon cantilever. This fabrication method was successfully demonstrated for two different probe designs. The design using an octahedron mould lead to a double tip, however, the design with a pyramidal mould (partially covered with oxide) lead to a single ultra-sharp tip. These probes were successfully mounted in a commercial AFM set-up. AFM images with a resolution less than 10 nm were obtained. The fabrication method is fully SOI compatible which allows for easy integration of an (electrostatic) MEMS actuator. This enables a higher frequency in the z-direction and makes video-rate AFM feasible.

VI – Acknowledgments

The authors would like to thank Mark Smithers for taking the SEM images.

References

[1] M.J. Rost, G.J.C. van Baarle, A.J. Katan, W.M. van Spengen, P. Schakel, W.A. van Loo, T.H. Oosterkamp, and J.W.M. Frenken. *Asian Journal of Control*, 11(2):110–129, 2009.

[2] G.E. Fantner, G. Schitter, J.H. Kindt, T. Ivanov, K. Ivanova, R. Patel, N. Holten-Andersen, J. Adams, P.J. Thurner, I.W. Rangelow, and P.K. Hansma. *Ultramicroscopy*, 106(8-9):881–887, 2006.

[3] S.R. Manalis, S.C. Minne, and C.F. Quate. *Applied Physics Letters*, 68(6):871–873, 1996.

[4] F. Levent Degertekin. In *Lasers and Electro-Optics Society, 2007. LEOS 2007. The 20th Annual Meeting of the IEEE*, pages 832 –833, 21-25 2007.

[5] K.E. Petersen. *Proceedings of the IEEE*, 70(5):420 – 457, may 1982.

[6] Z. Zhu and C. Liu. *Journal of Microelectromechanical Systems*, 9(2):252–261, 2000.

[7] Veeco Instruments. http://www.veeco.com/, 2010.